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VAPOUR PRESSURE DETERMINATION BY DIFFERENTIAL SCANNING CALORIMETRY (U)

(Presented at the 66th C.I.C. Chemical Conference and Exhibition June 5 - 8, 1983 by Ms. M.D. Gauthier)

by

M.D. Gauthier and S.J. Armour

PCN No. 13E10

December 1983

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PREFACE

This paper is the transcript of a talk presented at the 66th Chemical Conference and Exhibition of the Chemical Institute of Canada held in Calgary, Alberta, June 5 to 8, 1983 by Ms. M.D. Gauthier. It is hoped that the reader will remember when he is reading the paper that it was originally spoken rather than written and will forgive any deviations from accepted format.

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ABSTRACT

Various methods of measuring the vapour pressure curve of liquids and solids have been evaluated. The recently developed technique using differential scanning calorimetry (DSC) has been used to determine the vapour pressure of small quantities of materials. Vapour pressure data of a variety of liquids and solids, obtained using DSC, will be presented and compared with that determined by other methods. The advantages of this technique are also discussed.

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2. Schematic Diagram of DSC Vapour Pressure Instrumentation

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7. Typical Endotherm

8. Vapour Pressure Curve for Water, using Extrapolated Onset

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11. Vapour Pressure Data (log P vs 1/T)

12. Endotherm Displaying Pre-boiling Evapouration

13. Endotherm of a Contaminated Sample

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A vapour pressure curve can be determined by measuring the boiling point of a sample at 5 different pressures. Classically, this is very time consuming, and can require several millilitres of sample. Using thermal analysis, a curve can be determined in 4 – 5 hours, with less than 100 μL of material.

(FIGURE 1)

In thermal analysis, there are two specific techniques which can be used to measure vapour pressures. The first method is Differential Scanning Calorimetry (DSC) which is the measurement of **differential heat flow** between a sample and reference as a function of temperature as both are subjected to a controlled temperature program. The second technique is Differential Thermal Analysis (DTA) in which the **differential temperature** between the sample and reference are measured.

This talk will concentrate on DSC measurements.

The technique for using thermal analysis for vapour pressure measurements was first developed in the early 60's. Since then it's grown in popularity and there is now an American Society for Testing and Materials (ASTM) Committee investigating the use of thermal analysis as a standard method for vapour pressure determinations. The physical chemistry laboratory at DRES is involved in a round robin for measuring samples for the ASTM committee. At the present time, a preliminary standard procedure has been formulated and one set of samples has been analyzed by the members of the round robin. The committee is in the process of modifying the procedure before a second set of samples is distributed. Throughout this talk, the suggested ASTM procedures will be explained, along with any modifications we have incorporated in the course of our experiments.

Generally, the method involves heating a sample and a reference, in an enclosed cell, at a static pressure. The temperature is increased linearly past the boiling temperature of the sample and the resulting endotherm is recorded. The boiling point is noted and the vapour pressure is equal to the cell pressure during the run. This is repeated on fresh samples so that there are at least five sets of pressure, temperature data over as wide a pressure range as possible.

The method can be used to determine the vapour pressure curves of either liquids or solids, but it is restricted to either pure materials or azeotropic mixtures which do not undergo decomposition.

(FIGURE 2)

A schematic of the instrumentation used in DSC is as follows:

1. The principal part of the apparatus consists of a DSC cell and a cell base with a heater connected to a thermal analyzer. The thermal analyzer serves to control the heating rate and records the data. In our case, we have a DuPont pressure DSC cell, 910 Differential Scanning Calorimeter, and a 1090 Thermal Analyzer. For DTA, the same cell base and thermal analyzer would be used, but with a DTA cell.

2. A pressure measuring device and readout is also needed. We use an MKS Baratron pressure measuring system consisting of a sensor head which measures absolute pressure and a digital readout.

3. There is also a requirement for a vacuum source and a pressure source. For pressure, we use high purity nitrogen.

4. A ballast tank is required to help maintain constant pressure in the system during a run. We use an empty 20 lb propane cylinder which has a 12 L capacity.

The actual setup is shown in the next figure.

(FIGURE 3)

1. The DSC pressure cell is shown fully assembled in this figure. The pressure range for the cell is 0.01 to 53,000 torr. The cell base can be used with a pressure DSC cell, a regular DSC cell (2 torr-atmospheric), or a DTA cell. The thermal analyzer is microprocessor controlled with dual disk drives and a plotter. There is software available for the system for analyzing the data.

2. The MKS Baratron pressure measuring system used in these experiments has a 0 – 10,000 torr sensor head and a four place digital readout.

(FIGURE 4)

The details of the method are as follows. For DSC work a hermetically sealed pan, with a pinhole is used. The pans we use are anodized aluminum, approximately 6.5 mm in diameter, with a volume of 20 – 30 μL. Gold pans are available for those materials which react with aluminum.

The pinhole in the lid of the pan is very important. It allows the pressure to equilibrate between the interior of the sample pan and the cell. Otherwise there would be a pressure buildup inside the sample pan. The size of the pinhole is critical — if too large, effusion will occur and equilibrium conditions inside the sample pan will not be obtained. The recommended size of 0.38 to 0.64 mm allows the pinhole to approximate a short capillary tube, and as such the pan achieves equilibrium conditions. For substances with high vapour pressures, a smaller hole may be needed. For our work we used either a 0.34 mm hole or a 0.20 mm hole, achieving very good results with both. The holes were made with a Unimat drill, with either a twist drill bit or a pivot drill bit.

The sample size should be 1 – 3 μL or 1 – 3 mg, although this is somewhat sample dependant. If too large a sample is used, two things may occur: there may be

temperature gradients within the sample or there may be superheating and partial self-cooling. If too small a sample is used, a poor endotherm is obtained. We have used between 1 – 9 mg, with most samples around 5 mg. This seemed to be a good size with no indication that too much sample was present.

A dilutent is used with the sample in the pan. This has various uses: to increase the liquid surface area, to reduce vapourization prior to boiling by retention of the sample through surface tension, to act as nucleation sites for boiling (which minimizes super heating) and to provide a better match of heat transfer characteristics between the sample and reference pans. We use glass beads of 100 microns in size. Typically ~ 1 mg dilutent is used.

The reference pan is similar to that of the sample pan, and includes a pinhole and glass microbeads, but no sample.

Before the sample is put into a pan, the pan and lid are cleaned, and the pinhole is made. The dilutent is added next, then the sample. The pan and lid are sealed and the sample is now ready to go into the cell.

(FIGURE 5)

This figure shows a closeup of the interior of the pressure cell. The sample pan and reference pan are placed on a constantan disk through which the differential heat flow is measured. This is surrounded by a silver heating block. There is also a thermal radiation shield, and an outer pressure cylinder.

(FIGURE 6)

Before the pans are placed in the cell, the cell should be at the starting temperature. This is approximately 30°C below the expected boiling point. The cell is pressurized to the desired pressure and allowed to come to temperature equilibrium for approximately 1 minute. The run is then started, heating linearly at 10 deg/min, past the boiling point and the endotherm is recorded. Other heating rates in the range 10 – 20 deg/min may also be used. Too slow a heating rate, however, will result in rapid preboiling vapourization of the sample.

The pressure is held constant throughout the run. The vapour pressure of the sample is equal to the pressure in the cell. The pressure range of the method is from 0.075 torr to 53,000 torr, depending on the instrumentation used. With DTA, and DSC without a pressure cell, pressures above ambient cannot be used. In our lab, we are able to measure samples from 5 torr to 2,200 torr. In most classical methods, vapour pressure curves can be determined either below ambient pressure, or above ambient pressure, while with pressure DSC, a curve can be determined encompassing pressures both above and below atmospheric.

(FIGURE 7)

This figure shows a typical endotherm. Heat flow in milliwatts is measured along the y-axis and temperature in degrees celsius is recorded on the x-axis. The run was started ~ 20°C below the boiling point. The initial deflection indicates a lag while the cell goes from an isothermal temperature to the heating mode. Then there is a flat baseline until boiling occurs. Ideally the sample should undergo isothermal boiling, with a vertical slope on the leading edge. With DSC however, there is some temperature rise, usually between 2 – 10 degrees, as the sample boils. The downward peak indicates an endothermic process as heat is being absorbed by the sample as it boils. After the sample has evapourated, the curve flattens out and the sample pan is essentially the same as the reference pan.

The boiling point in DSC is taken as the extrapolated onset temperature. This is obtained by drawing a tangent from the steepest part of the curve and intersecting it with the extrapolated baseline. This is done by hand, then the DuPont 1090 software is used to determine the exact temperature. The peak temperature is also recorded. Presently there is some controversy in the ASTM committee over which temperature to record as the boiling temperature. Traditionally it has been the extrapolated onset for DSC, however the results from the initial round robin tests indicate that the peak temperature may be more valid. We have examined both temperatures during our experiments to determine which one compares better with published data.

(FIGURE 8)

Once all the pressure temperature data points are determined, a plot of log P vs 1/T (in degrees Kelvin) is made. For the ASTM procedure, the test is done

twice, on two different days and both sets of data are reported. This graph shows the variability of a water sample on two different days, using the extrapolated onset temperature. The two lines agree to within 0.3% at the high pressure end and to within 0.1% at the low end.

(FIGURE 9)

The variability in the peak data for the same two days is shown in this figure. Again the data is very reproducible, with a difference of 0.1% at high pressures and 0.5% at low pressures.

(FIGURE 10)

This figure shows the relationship of the experimental data with the literature data for water. For the extrapolated onset temperatures, the agreement of the curves with the literature data is within 0.4% at the high and low pressures while for the peak temperatures the agreement is 1.5% at the high pressures and 4.5% at the low pressures. Both extrapolated onset and peak temperatures give good straight lines with correlation coefficient (R^2) better than 0.998 for both lines.

(FIGURE 11)

We have determined the vapour pressure curves for several other materials, both liquids and solids. Data is presented here for hexane, napthalene, benzoic acid and methyl salicylate. For all materials, duplicate runs were made. In each case, plots of log P against 1/T were linear over the pressure range measured (20 – 2100 torr) with good correlation coefficients for both the extrapolated onset temperatures and the peak temperatures.

A comparison of the data with literature values indicates that for each compound tested the extrapolated onset temperature was closer to the expected value than was the peak temperature, and was within ± 1% of the literature value.

The peak temperatures were within ± 5% for each compound. Using our experimental setup, it appears that the extrapolated onset temperature gives the better data and should be used preferentially over the peak temperature whenever possible.

(FIGURE 12)

There are some instances where it is not possible to measure the extrapolated onset temperature. If equilibrium conditions are not achieved within the sample pan, preboiling evapouration may occur. This results in a gradual decrease in the baseline, with little or no isothermal boiling. In this figure, the material is benzoic acid. When the sample was placed in the cell, the starting temperature was not high enough for all the solid to melt, and temperature equilibrium was not achieved initially. There was a continual loss of sample through sublimation. The resulting onset temperature was 13% lower than expected, while the peak temperature was within 1.3%. Preboiling evapouration may also occur with highly volatile materials, either liquid or solid, or if the sample container does not approximate a capillary. The achieving of capillary equivalent conditions is a particular problem with DSC. With DTA the instrumental configuration is such that capillary equivalent conditions are more closely approximated.

(FIGURE 13)

The use of DSC to measure vapour pressure is limited to pure substances or azeotropic mixtures which do not undergo decomposition prior to or during the isothermal vapourization. This is a sample of benzoic acid with a contaminant, run at 108 torr. Thermal analysis is very useful in this case as the impurity or decomposition is easily detected in the thermogram. Usually there are multiple peaks, non-isothermal boiling or a non-linear vapour pressure function. In this case, the boiling point can not be determined.

(FIGURE 14)

There are several advantages to using DSC or DTA for vapour pressure measurements, over other methods.

1. An extremely small sample size is required;
2. no pretreatment of the sample is necessary;
3. impurities or decomposition in the sample can be readily detected;
4. the method can be used for liquid or solid material;

5. the sample is not heated at the boiling point for prolonged periods, which would increase the possibility of decomposition;
6. with a pressure DSC cell, determinations can be made above and below ambient pressure;
7. it is a simple, rapid method: a complete curve can be determined in less than 5 hours;
8. the cell can be easily isolated for work with hazardous materials.

In conclusion, pressure DSC can be used to measure vapour pressure curves for liquids and solids. The data is in good agreement with published values. We have found that in all cases the extrapolated onset temperature was closer to the published data than was the peak temperature, although the peak values were within ± 5%. In samples that show preboiling evapouration, the peak temperature may be used as a good approximation to the actual temperature.

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4. A.A. Van Cooren and B.W. Muller, *Thermochimica Acta,* **49** (1981) p. 151 – 161.

VAPOUR PRESSURE MEASUREMENTS USING THERMAL ANALYSIS

DIFFERENTIAL SCANNING CALORIMETRY (DSC)

- MEASUREMENT OF DIFFERENTIAL HEAT FLOW BETWEEN A SAMPLE AND REFERENCE MATERIAL AS A FUNCTION OF TEMPERATURE WHILE BOTH ARE SUBJECTED TO A CONTROLLED TEMPERATURE PROGRAM.

DIFFERENTIAL THERMAL ANALYSIS (DTA)

- MEASUREMENT OF TEMPERATURE DIFFERENCE BETWEEN A SAMPLE AND REFERENCE MATERIAL, AS A FUNCTION OF TEMPERATURE, WHILE BOTH ARE SUBJECTED TO A CONTROLLED TEMPERATURE PROGRAM.

FIGURE 1

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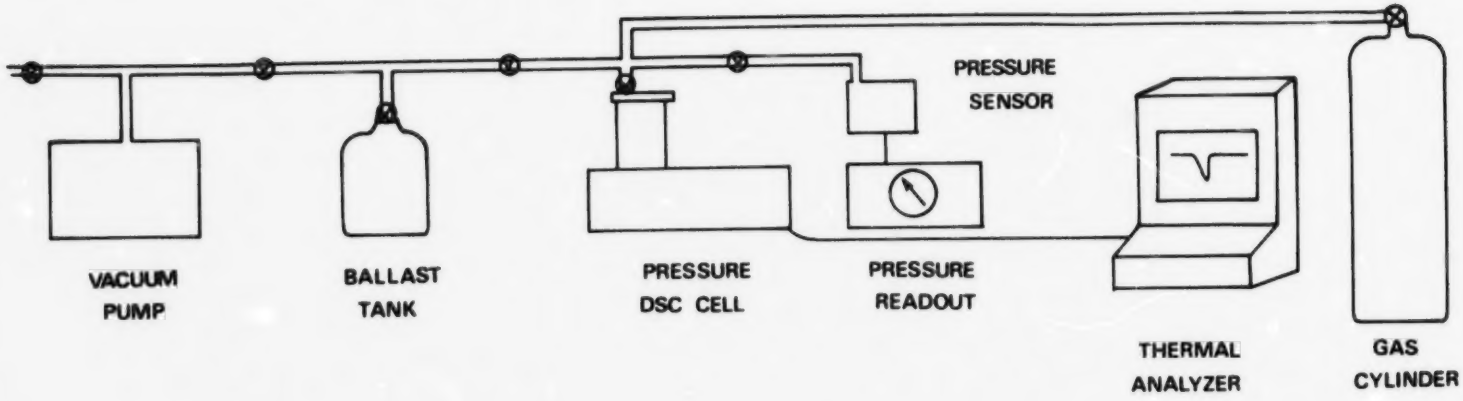

SCHEMATIC DIAGRAM OF DSC VAPOUR PRESSURE INSTRUMENTATION

FIGURE 2

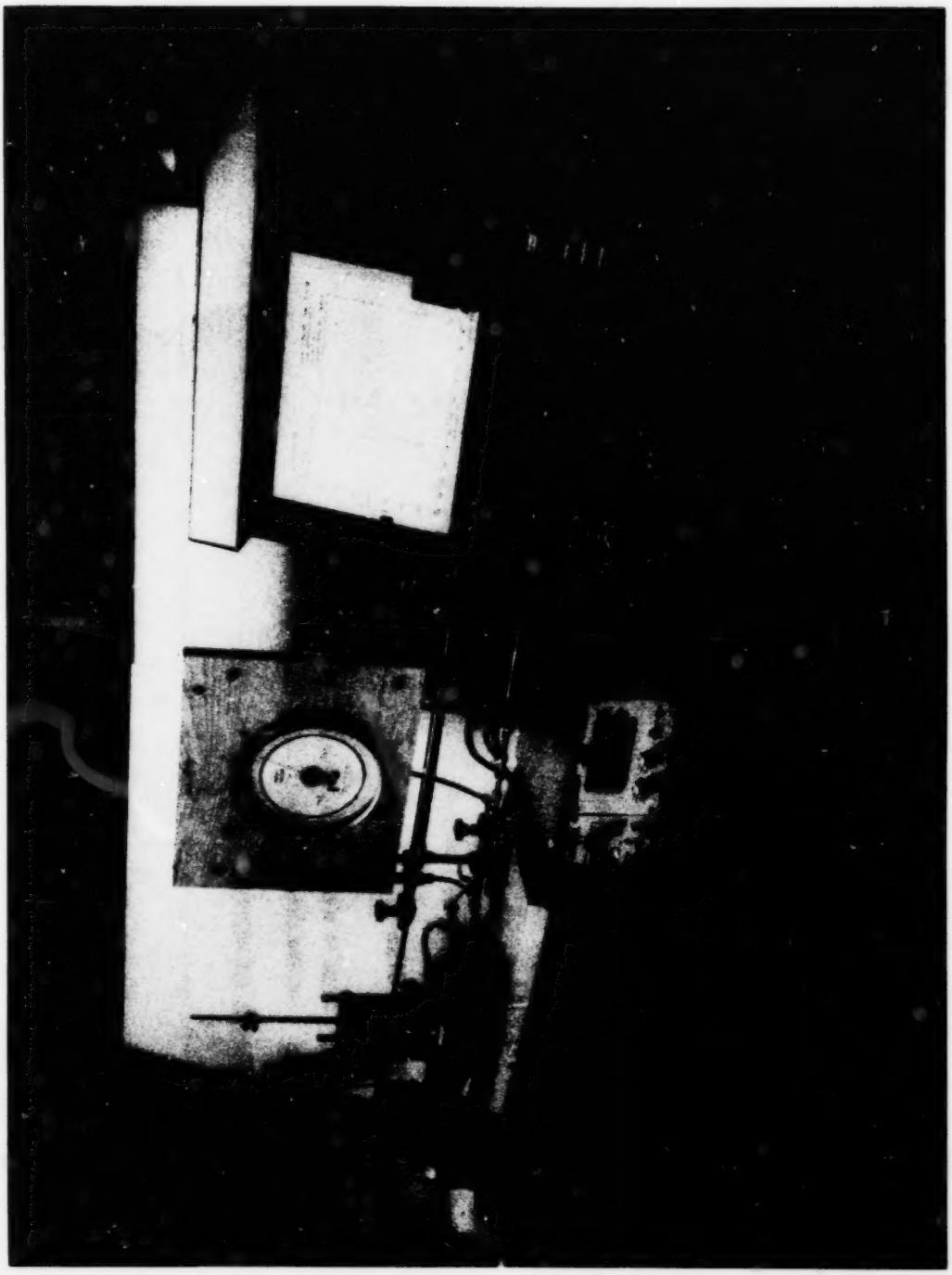

FIGURE 3

METHOD FOR VAPOUR PRESSURE MEASUREMENTS USING DSC

SAMPLE CONTAINER

- HERMETICALLY SEALED PAN WITH PIN HOLE (0.38 to 0.64 mm DIAMETER)

SAMPLE SIZE

- 1 to 3 μL or 1 to 3 mg

DILUTANT

- GLASS μBEADS, CARBORUNDUM, OR POWDERED ALUMINUM OXIDE (APPROXIMATELY 1 mg)

REFERENCE

- HERMETICALLY SEALED PAN, WITH PIN HOLE AND DILUTANT

FIGURE 4

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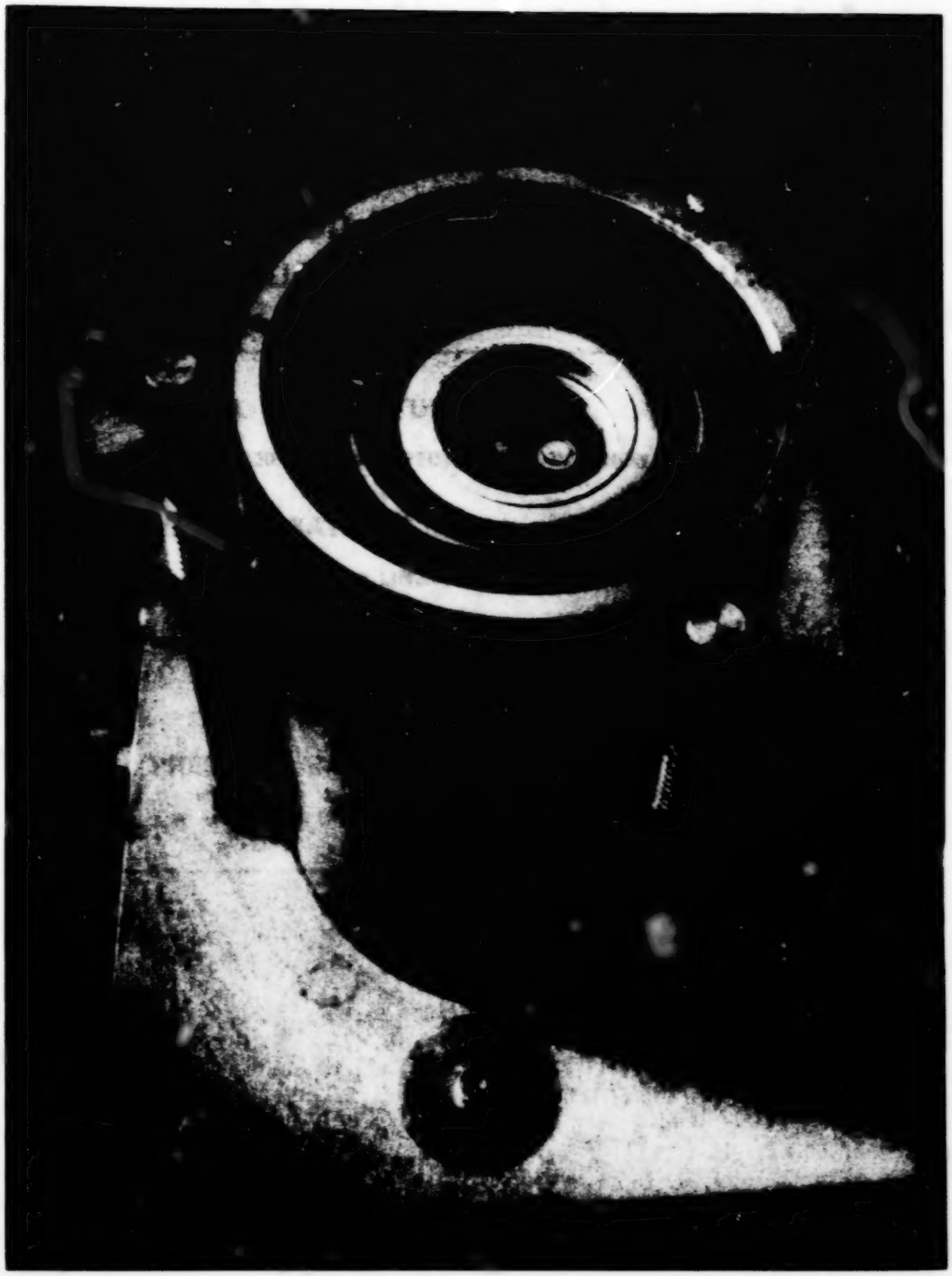

FIGURE 5

METHOD FOR VAPOUR PRESSURE MEASUREMENTS USING DSC (Cont'd)

INITIAL TEMPERATURE

- 30° BELOW EXPECTED VAPORIZATION TEMPERATURE

HEATING RATE

- 10 deg/min LINEARLY

PRESSURE

- STATIC THROUGHOUT RUN

PRESSURE RANGE

- 7.5×10^{-2} TORR TO 5.3×10^{4} TORR

FIGURE 6

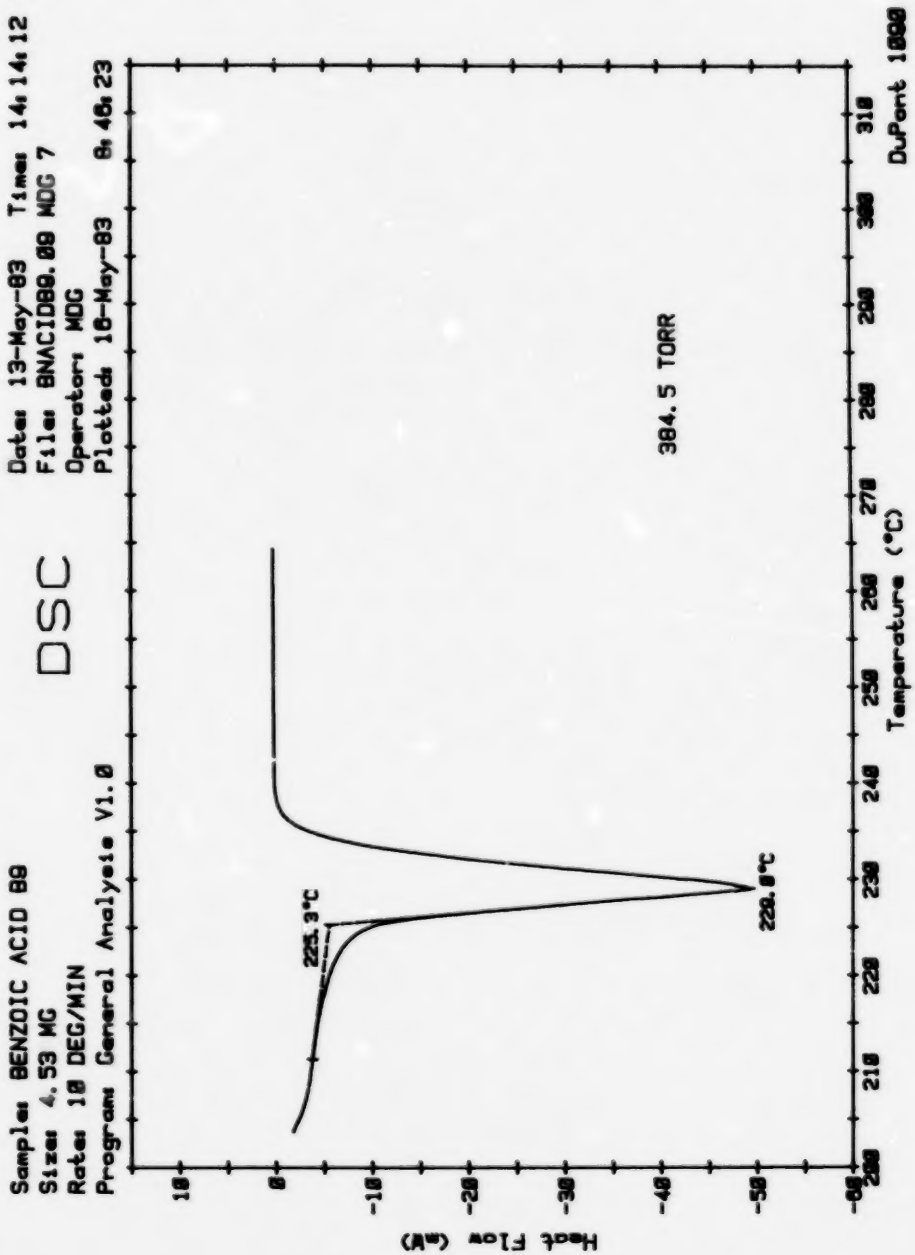
Sample: BENZOIC ACID 88
Size: 4.53 MG
Rate: 10 DEG/MIN
Program: General Analysis V1.0
DSC
Date: 13-May-83 Time: 14:14:12
File: BNACID88.00 MDC 7
Operator: MDC
Plotted: 16-May-83 8:46:23
384.5 TORR
225.3°C
228.8°C
Heat Flow (mW)
Temperature (°C)
DuPont 1000

FIGURE 7

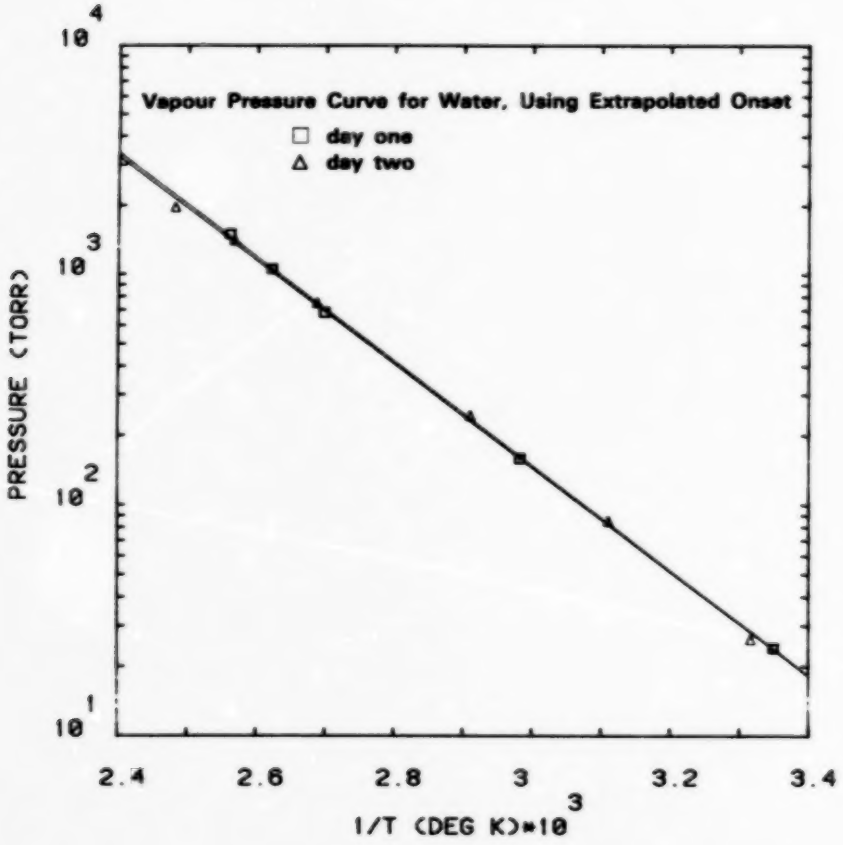
Vapour Pressure Curve for Water, Using Extrapolated Onset
day one
day two
PRESSURE (TORR)
10^4
10^3
10^2
10^1
2.4
2.6
2.8
3
3.2
3.4
1/T (DEG K)*10^3

FIGURE 8

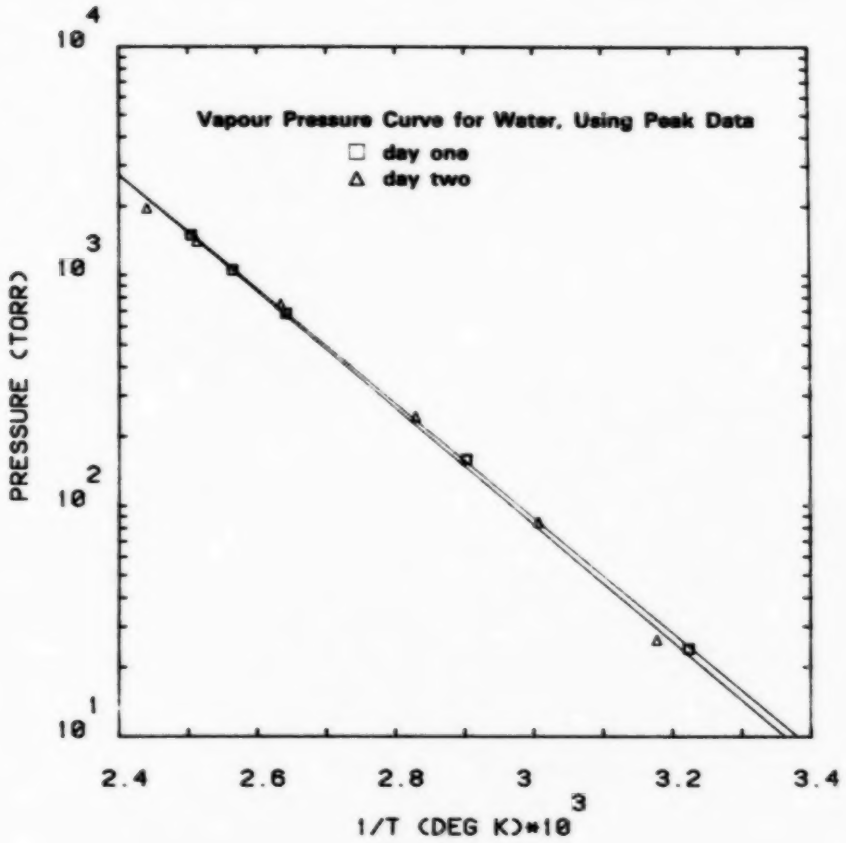
Vapour Pressure Curve for Water, Using Peak Data
□ day one
△ day two
PRESSURE (TORR)
10^4
10^3
10^2
10^1
2.4
2.6
2.8
3
3.2
3.4
1/T (DEG K)*10^3

FIGURE 9

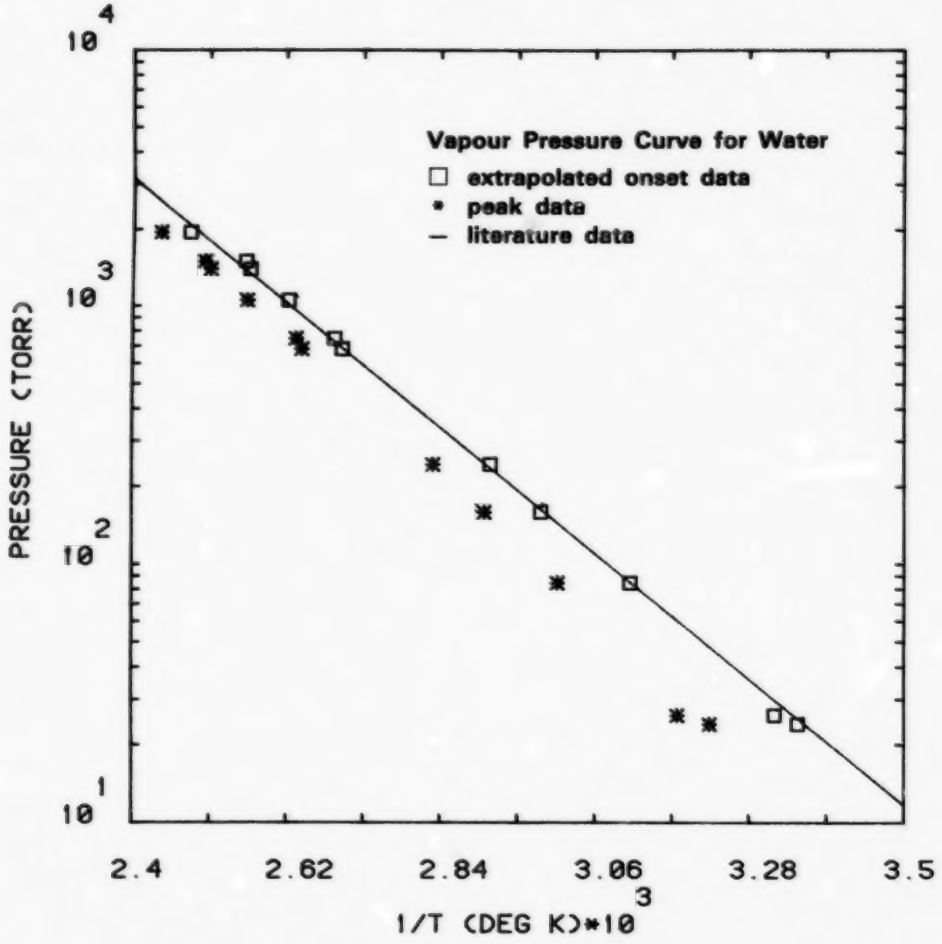
Vapour Pressure Curve for Water
extrapolated onset data
peak data
literature data
PRESSURE (TORR)
10^4
10^3
10^2
10^1
2.4
2.62
2.84
3.06
3.28
3.5
1/T (DEG K)*10^3

FIGURE 10

VAPOUR PRESSURE DATA (log P vs 1/T)

SAMPLE	R^2		% ERROR WITH LITERATURE DATA			
	EXTRAPOLATED ONSET	PEAK	EXTRAPOLATED ONSET		PEAK	
			HIGH P	LOW P	HIGH P	LOW P
WATER	0.999	0.998	- 0.2	+ 0.4	+ 1.5	+ 4.5
HEXANE	0.997	0.998	- 0.7	+ 0.4	+ 0.7	+ 1.4
NAPTHALENE	0.999	0.998	+ 0.2	+ 0.4	+ 0.6	+ 2.6
BENZOIC ACID	0.995	0.992	+ 0.6	- 0.9	+ 1.6	+ 0.7
METHYL SALICYLATE	0.999	0.998	+ 0.4	- 0.1	+ 1.1	+ 1.2

FIGURE 11

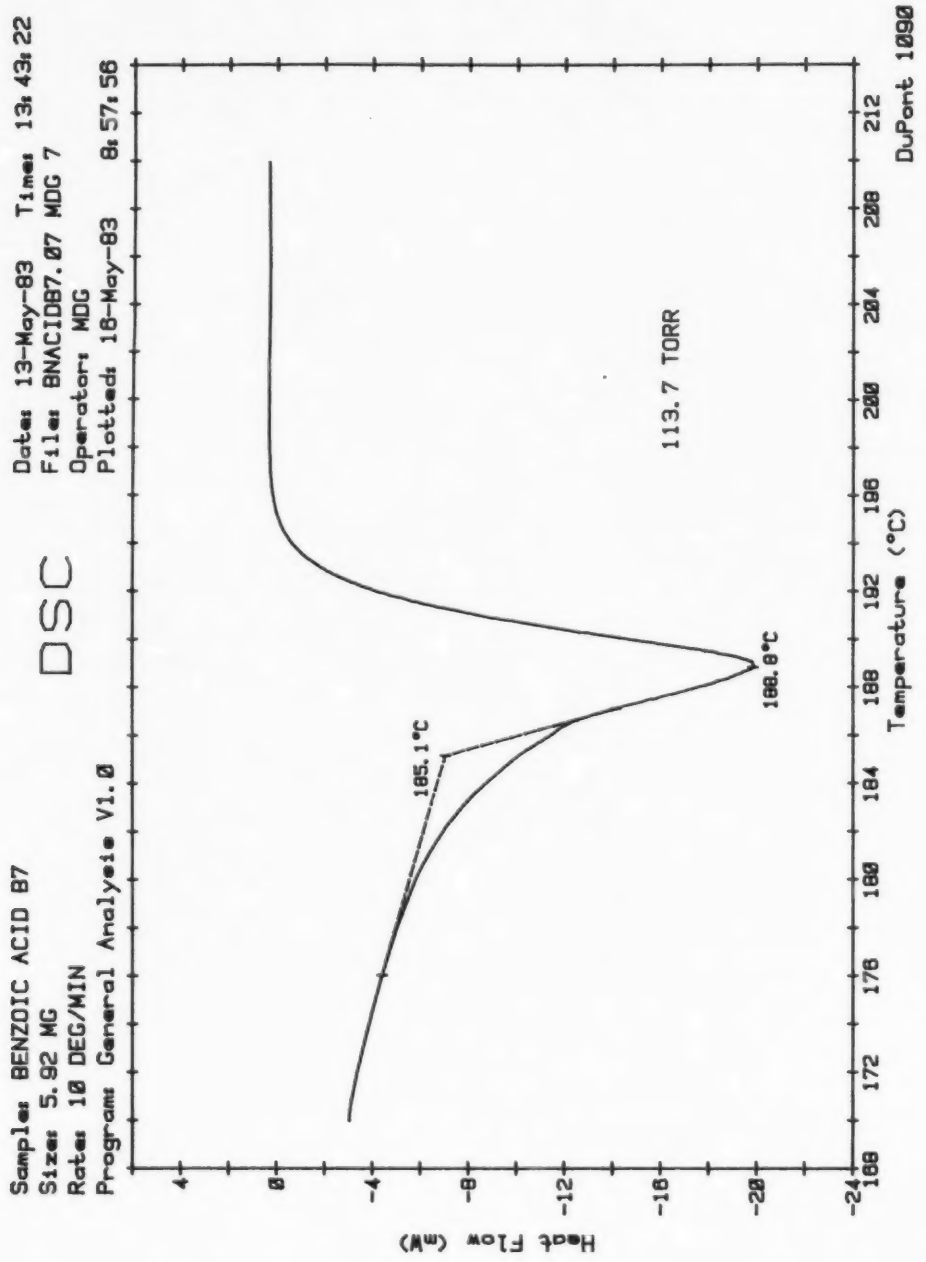
Sample: BENZOIC ACID B7
Size: 5.92 MG
Rate: 10 DEG/MIN
Program: General Analysis V1.0
DSC
Date: 13-May-83 Time: 13:43:22
File: BNACIDB7.07 MDG 7
Operator: MDG
Plotted: 18-May-83 8:57:56
113.7 TORR
185.1°C
188.8°C
Heat Flow (mW)
Temperature (°C)
DuPont 1090

FIGURE 12

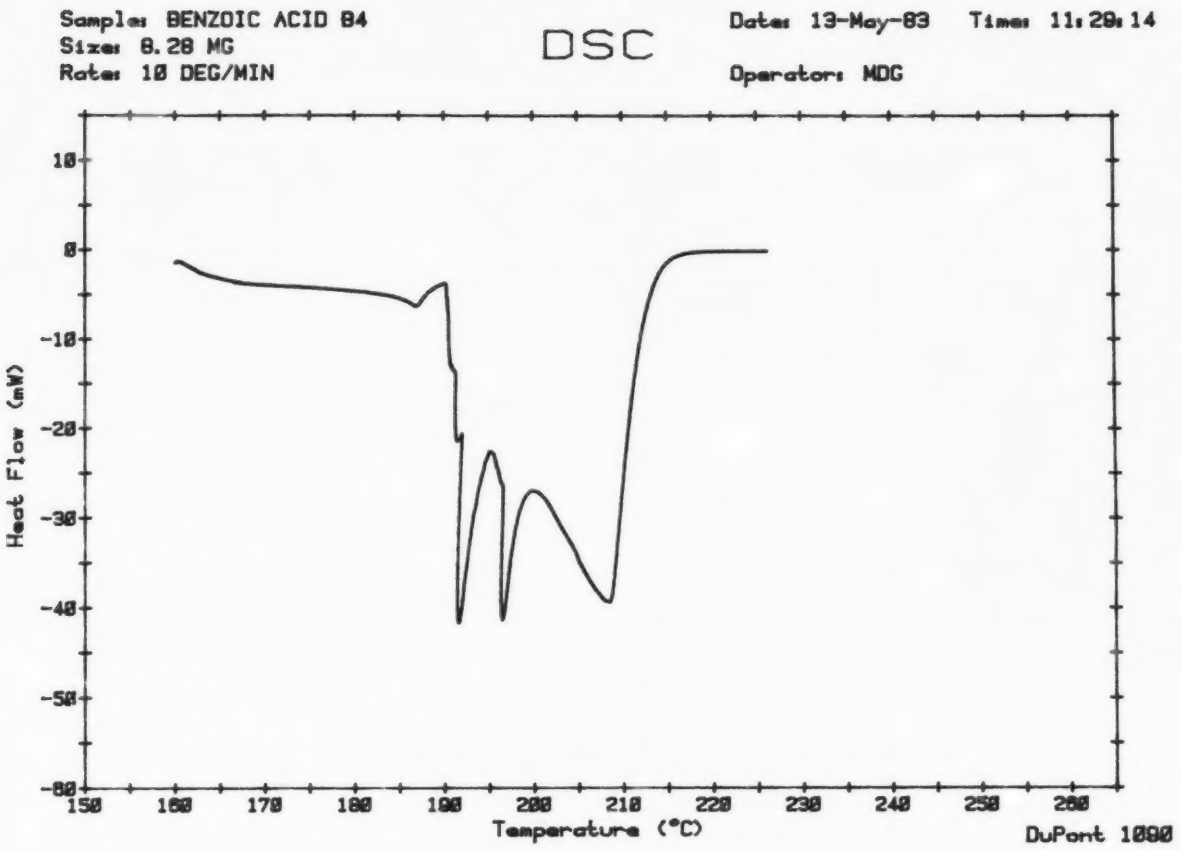
Sample: BENZOIC ACID B4
Size: 8.28 MG
Rate: 10 DEG/MIN
DSC
Date: 13-May-83 Time: 11:29:14
Operator: MDG
10
0
-10
-20
-30
-40
-50
-60
Heat Flow (mW)
150 160 170 180 190 200 210 220 230 240 250 260
Temperature (°C)
DuPont 1090

FIGURE 13

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ADVANTAGES OF DSC OR DTA VAPOUR PRESSURE METHOD

1. EXTREMELY SMALL SAMPLE SIZE
2. NO PRETREATMENT OF SAMPLE
3. DECOMPOSITION OR IMPURITIES IN SAMPLE READILY OBSERVED
4. APPLICABLE TO SOLIDS AND LIQUIDS
5. NO PROLONGED HEATING OF THE SAMPLE AT THE BOILING POINT
6. WITH PRESSURE DSC, PRESSURES ABOVE AND BELOW AMBIENT CAN BE USED
7. SIMPLE, RAPID METHOD (< 5 hours for a complete curve)
8. CELL CAN BE EASILY ISOLATED FOR USE WITH HAZARDOUS MATERIALS

FIGURE 14

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13. ABSTRACT

Various methods of measuring the vapour pressure curve of liquids and solids have been evaluated. The recently developed technique using differential scanning calorimetry (DSC) has been used to determine the vapour pressure of small quantities of materials. Vapour pressure data of a variety of liquids and solids, obtained using DSC, will be presented and compared with that determined by other methods. The advantages of this technique are also discussed.

KEY WORDS

Vapour Pressure

Differential Scanning Calorimetry (DSC)

Thermal Analysis

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